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Project NY 030 019-1.04  
Technical Note N-328

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CORROSION INHIBITORS FOR LITHIUM CHLORIDE SOLUTIONS

C. V. Brouillette

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FEB 10 1958  
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U. S. Naval Civil Engineering Research and Evaluation Laboratory  
Port Hueneme, California

Project NY 030 019-1.04  
Technical Note N-328  
CORROSION INHIBITORS FOR LITHIUM CHLORIDE SOLUTION  
C. V. Brouillette

~~OBJECT OF PROJECT~~

To ascertain the most suitable extinguishment available for Class A fires and the best methods of building protection under the adverse conditions found in advanced Arctic and Antarctic bases,

~~OBJECT OF SUBPROJECT~~

To determine container materials or corrosion inhibitors, or both, compatible with water solutions of lithium chloride for use as a fire extinguishing agent at low temperatures.

OBJECT OF REPORT

To present the results of studies and experimentations on the effects of various chemicals toward lessening the corrosive attack of 24 percent aqueous solutions of lithium chloride upon fire extinguishers and the materials from which they are fabricated.

RESULTS

The 2-1/2 gallon copper fire extinguisher containing sodium dichromate-oxalic acid inhibitor was protected to the greatest extent from the 24 percent lithium chloride solution.

The stainless steel fire extinguishers were attacked at the water line and at welds. Intergranular and crevice corrosion was so severe that leaks developed in areas adjacent to the welds.

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#### SUMMARY

The results of the studies and experimentations showed that in the presence of inhibitors the stainless steel test panels did not corrode but that the stainless steel fire extinguishers corroded severely. The corrosion in the extinguishers was confined almost entirely at the welds and crevices which were not present on the test panels.

The series of copper panels tested showed greater corrosion losses than did the stainless steel or brass series. Yet, the copper extinguishers, because of the lining of lead, appeared to be satisfactory for use with inhibited lithium chloride solutions. In instances where copper surfaces were exposed through the lead lining, corrosion did occur.

The series of brass panels tested in the presence of the sodium dichromate-oxalic acid inhibitor or the sodium dichromate citric acid inhibitor resisted corrosion better than did the series of copper panels. No brass fire extinguishers were available for use in the full scale corrosion tests. However, such extinguishers manufactured of drawn brass are commercially available, are heavily lead lined, contain no solder, and are pressure tested to 500 psi. Inasmuch as pressures near 500 psi are possible, if a restriction occurs in the delivery hose, the brass extinguishers would be more desirable than the copper extinguishers which are tested for 350 psi pressure.

## INTRODUCTION

Investigations under the subproject title "Low Temperature Fire Extinguishing Agents"<sup>1</sup> resulted in the recommendation that water solutions of 24 percent lithium chloride or 10 percent lithium chloride-20 percent calcium chloride be used for class A first aid fire extinguishers. Lithium chloride is preferred to the lithium chloride-calcium chloride combination because it had a lower eutectic with water. Under subproject NY 030 019-1.04, "Investigation of Effective Inhibitors for Lithium Chloride Solutions," the development of effective corrosion inhibitors to protect the fire extinguisher from the corrosive action of the salt solution was also undertaken.

The metals commonly used in the construction of fire extinguishers include copper, brass, and stainless steel. These are often lead lined and soldered at seams. Thus, the inhibitor which is incorporated in the lithium chloride solution must be effective in the presence of electrolytic couples of these metals.

Preliminary laboratory tests favored the use of stainless steel with the sodium dichromate inhibited solution.<sup>2</sup>

## LABORATORY PROCEDURE

Small metal test panels (3 in. (inch) by 1/2 in. by 1/8 in.) were prepared from copper, brass, and stainless steel. One-third of the metal test panels were prepared with a thin strip of 50-50 tin-lead solder down one edge and one-third were prepared with a thin strip of silver solder (Solvaloy 50) down one edge. Thus, a total of nine metals and metal combinations were tested. The test panels were weighed, placed in 30 ml (milliliter) test tubes, approximately 10 ml of 24 percent lithium chloride solution containing the test inhibitor was added, and the stoppered test tube was placed in a constant temperature cabinet. The 10 ml of inhibited salt solution was sufficient to cover the lower two-thirds of the metal test panel. One-half of the tests were conducted at 100°F (degree Fahrenheit) and one-half at 140°F. The tests were continued for 2 months, then the contents of the test tubes were given a careful visual inspection, the weight loss of the panels was determined, and the cleaned panels were inspected visually.

During the first visual inspection, the amount of sediment which was formed in the salt solution and the areas of

corrosion attack on the metal test panels were recorded. These areas of attack were: (a) the panel area above the liquid surface, (b) the panel area below the liquid surface, and (c) the area at the liquid-air interface. During the second visual inspection, the amount of attack on the metal of the panel and on the soldered area was noted.

The test panels were removed at the end of the test period and cleaned by use of water and a scrub brush, then by dipping for approximately 10 sec (seconds) in warm concentrated hydrochloric acid, rinsing with water and lightly cleaning with a rubber ink eraser. The loss of metal, except from solder, was found to be negligible by this method of cleaning. The panels were then dried and the weight loss determined and calculated to mdd (milligrams per square decimeter per day). An error in weighing of any test panel of 1 mg (milligram) was the equivalent to a corrosion loss of approximately 0.07 mdd. Cleaning losses of the panels containing the soft tin-lead (50/50) solder were slightly higher than for the other panels. Cleaning losses varied from 0.125 mdd for the stainless steel test panels to 0.70 mdd for the stainless steel containing tin-lead solder along one edge. Corrections were made for the cleaning losses in the tabulated results.

In order to economize on time and materials, a quick screening method was used to eliminate those inhibitors which do not materially affect the attack by the lithium chloride solution. The copper test panels were generally the most severely attacked by the 24 percent lithium chloride solution even in the presence of the inhibiting chemicals. For this reason a copper test panel containing a strip of tin-lead (50/50) solder was chosen for the screening tests. The screening tests were conducted as described above for 3 days at 140°F. At the end of this time, if no corrosive attack was evident, the inhibitor was used for the full scale 2 months test in both the 100°F and the 140°F test environments.

## RESULTS

Tests of 238 potential inhibitors were made by means of a screening test using a copper panel containing tin-lead solder along one edge. Fifteen inhibitors gave sufficient protection to warrant further testing in the full scale test. Sodium dichromate appeared to be the most satisfactory individual inhibitor, but in most instances, it gave better protection when used in combination with either oxalic or citric

acid. For very short periods of time a piece of tin in contact with the copper panel and in the presence of an acidic organic inhibitor protected the soldered copper test panels. A bright tin plate formed on the surface of the panel. This action prevented the attack on the solder for the 3 day screening tests but failed to protect the soldered copper test panels during the 2 month evaluation period.

Table I gives corrosion losses of the 9 test panels in each of the 15 chemical inhibitors and 2 sets of controls which were tested for 2 months at 140°F. The controls, especially the copper and brass panels in contact with tin-lead solder, showed quite a wide difference in corrosion losses at 140°F. This was attributed to the nonuniformity in the thickness of the solder coating placed along the edge of these panels. The solder was preferentially attacked and those panels having the thicker coating of solder showed the greater corrosion loss. The inhibitor, composed of 0.2 percent oxalic acid and 0.5 percent sodium dichromate, gave the greatest reduction in corrosion of any of the systems tested. The inhibitor composed of 0.2 percent citric acid and 0.5 percent sodium dichromate gave equal protection for the brass test panels, slightly better protection for the copper-tin-lead soldered panel, and slightly less protection for the stainless steel panels containing solder. The stainless steel-soldered (tin-lead) test panel was protected best by sodium dichromate (0.5 percent) alone.

The inhibitors composed of triethanol amine, diethanol amine, or diethylethanol amine in conjunction with sodium dichromate gave protection to the stainless steel series of test panels equal to that of the sodium dichromate-oxalic acid inhibitor, but less than sodium dichromate alone. These inhibitors also protected the brass series of panels but the copper panels were given only partial protection.

Table II gives the corrosion losses in mdd for the metal test panels which were exposed for 2 months at 100°F. The corrosion losses observed on the controls, exposed at 100°F, are 34 to 75 percent less than the losses resulting at 140°F. The sodium dichromate-oxalic acid (or citric acid) gave the greatest amount of protection to the test panels at the 100°F temperature. The amines mentioned above gave satisfactory protection to the stainless steel panels and partial protection to the brass panels. At the lower temperature the amine inhibitors increased corrosion of the copper series of test panels by formation of a soluble blue copper-amine complex.



From the results of the accelerated laboratory tests using small metal test panels, the sodium dichromate-oxalic acid inhibitor appears to be the most satisfactory. The series of brass and stainless steel metal test panels showed greater corrosion resistance in the presence of sodium dichromate-oxalic acid inhibitor than did copper.

#### TESTS ON 2-1/2 GALLON FIRE EXTINGUISHERS

Since the inhibitor tests conducted in the test tubes on small metal test panels were not conclusive, final comparisons of the promising inhibitors were made using 2.5 gal (gallon) fire extinguishers. Two types of extinguishers were available, one manufactured of stainless steel and one of copper. The seams on the stainless steel extinguishers were electrically welded. The vertical seam on the copper extinguishers was held by copper rivets and tin-lead solder while the seams around the top and bottom were merely soldered. A protective lining of lead completely covered the inside of these extinguishers. Three stainless steel and three copper extinguishers were used for the final tests. One extinguisher of each type was filled with: (1) a sodium chromate inhibited 24 percent solution of the lithium chloride supplied by the NRL (Naval Research Laboratory), Washington, D. C., (2) a 24 percent solution of lithium chloride containing 0.5 percent sodium dichromate and 0.2 percent oxalic acid, and (3) a 24 percent solution of lithium chloride containing 0.5 percent sodium dichromate.

The extinguishers containing the salt solutions were placed in a constant temperature cabinet held at 140°F (60°C) for 2 months or until failure. At the end of this period the extinguishers were cut open, the inner surfaces inspected, and the salt solutions examined for evidences of corrosion products.

#### Stainless Steel Extinguishers

All three stainless steel extinguishers were severely corroded around the welded seams at the top and bottom with only light corrosion occurring along the vertical seams, (figures 1 through 4). The attack along the vertical seam of extinguisher 1 containing the NRL sodium chromate inhibitor was greater than that along the vertical seams of Extinguishers 2 or 3 which contained sodium dichromate-oxalic acid or sodium dichromate as the inhibitors.

It will be noted in figures 5 and 6 that the extinguishers are constructed so that a small crevice is formed between the upper edge of the bottom section and the body of the extinguisher. This same condition occurs at the top seam. In extinguisher 3 (figure 2) the rounded bottom is inverted and little if any corrosion was evident.

Spectrographic analysis of samples of stainless steel taken from extinguishers 1, 2, and 3 showed all the alloys to be the same and most likely AISI 303.

Both crevice corrosion (figure 5) and intergranular corrosion (figure 7) occurred at the welded and lapped type of construction found on the stainless steel extinguishers. According to Uhlig<sup>3</sup> even certain gasketing material in contact with stainless steel will cause crevice corrosion. Intergranular corrosion was observed in the metal proper adjacent to the weld (figure 7). Heat treatment resulting from the welding had caused the metal adjacent to the weld to become anodic to the weld. Corrosion appeared to be most severe at areas where the weld and crevice were in close proximity.

Stainless steel extinguisher 1, which contained the NRL sodium chromate inhibitor, rusted so rapidly in this area that three small holes appeared after 3 weeks accelerated exposure at 140°F (figures 1, 3, and 5). At the end of 4 weeks, extinguisher 2 containing sodium dichromate-oxalic acid inhibitor was corroded through at one place (figures 1, 4, and 6).

The corrosion products found in the lithium chloride solution taken from the stainless steel extinguishers were mostly red rust. It will be noted that the top inside area of extinguisher 1 had very little corrosion (figure 8). This extinguisher, prior to its use in the accelerated corrosion test, had been used to test the NRL slow burning propellant. A thin film of a greasy carbon residue had formed over the upper surfaces of the extinguisher and offered considerable protection against the lithium chloride solution. This greasy film could be wiped off quite easily with a rag.

#### Copper Extinguishers

Extinguisher 4 (figure 9) contained the NRL sodium chromate inhibitor, extinguisher 5 (figure 10) contained sodium dichromate-oxalic acid inhibitor, and extinguisher 6 (figure 11) contained sodium dichromate inhibitor.

The corrosion of the copper extinguishers was confined mostly to the solder along the seams. The copper extinguishers employed in this test had previously been in use and could not be properly cleaned before starting the test. The rough appearing surface of the side walls and bottoms was hard water scale and was present at the start of the test, but the additional scale and corrosion products at the top were formed during the course of the test. Complete removal of the hard water scale on the inside of the extinguishers at the end of the test period showed no attack on the uniform film of lead with which the extinguisher had been coated. The lithium chloride solution emptied from extinguishers 4 and 6 contained considerable amounts of corrosion products in the form of sediment; however, the salt solution emptied from extinguisher 5 was practically free of sediment, indicating little if any corrosion had occurred.

After cutting the extinguishers in half, large clumps of solder were found along the vertical seam of extinguisher 6 (figure 11). A similar situation was encountered in extinguisher 4 (figure 9). The clumps of solder were covered with crystals of lead chromate and were soft and porous, (figure 12). Some clumps had separated from the sides of the extinguishers and collected as sediment on the bottom. Analysis showed the porous material and the sediment to be mostly lead together with a small amount of tin, copper and lead chromate. Thus, corrosion attack on these clumps of solder had caused dissolution of the tin leaving a soft, porous lead residue.

Careful inspection of the inside surfaces of the three extinguishers showed areas of slight pitting attack in extinguisher 6. This pitting was at breaks in the very thin lead coating where the underlying copper had been exposed. It was evident that this coating was thin at the start of the test because only a slight amount of lead chromate was found in the corrosion products at the end of the test. The corrosive attack in extinguishers 4 and 6 was almost entirely on the clumps of excess solder found along the vertical seams. Extinguisher 5 showed little or no attack and contained no loose corrosion products. The lead coating inside this extinguisher was continuous with no areas of exposed copper. Spectrographic analysis of solder samples taken from the top, bottom and vertical seams showed them to be tin-lead solder containing nearly equal proportions of the two metals.

### Results

Copper extinguisher 5 (figure 10) which contained sodium dichromate-oxalic acid inhibitor, appeared to be satisfactorily

protected during the 2 month accelerated corrosion test. The soldered surfaces showed no signs of attack. The corrosion of extinguisher 4 (figure 9) which contained the NRL sodium chromate inhibitor and extinguisher 6 (figure 11) which contained sodium dichromate inhibitor was confined almost entirely to the clumps of solder along the seams.

The lead coating inside the copper extinguisher at the end of the 2 month accelerated test showed no evidences of corrosion attack. In extinguisher 6 the lead lining was very thin with some copper surfaces exposed. These copper surfaces were pitted slightly.

None of the stainless steel extinguishers were protected satisfactorily from corrosion during the 2 month accelerated test. Two of the three extinguishers did not survive the 2-month test. The crevices at the joints and at the welded seams were the focal points of attack. Attack was aggravated at the welds by intergranular corrosion. The inverted bottom of extinguisher 3 (figure 2) did not produce the type of crevice which was found in extinguishers 1 and 3 (figures 5 and 6). Only a slight amount of corrosion appeared around the inside bottom seam of this extinguisher. Intergranular corrosion occurred along all welded seams but was more pronounced on extinguisher 1 (figures 5 and 7).

Pressure tests at NRL using the slow burning propellant in a 2-1/2 gal fire extinguisher measured pressures up to 500 psi (pounds per square inch) if the flow of extinguishing fluid is restricted. Under normal operation the pressure remained below 100 psi.

#### CONCLUSIONS

1. The sodium dichromate-oxalic acid inhibitor gave satisfactory protection to the lead lined copper fire extinguisher.
2. The NRL sodium chromate inhibitor and the NAVCERELAB sodium dichromate inhibitor permitted considerable corrosion to solder in the copper extinguishers. The lead lined surfaces inside the copper extinguishers appeared to be satisfactorily protected by these inhibitors.
3. Severe corrosion occurred in the stainless steel extinguishers especially along crevices and adjacent to welds. Stainless steel fire extinguishers are not satisfactory for use with 24 percent lithium chloride solution.

4. Brass was shown to be more resistant to corrosion in inhibited 24-percent lithium chloride solution than copper. Because brass fire extinguishers are pressure tested to 500 psi they would be preferred to the copper extinguishers for use with the NRL propellant charge.

#### RECOMMENDATIONS

1. Heavily lead lined drawn brass fire extinguishers, containing no solder, are recommended for use with 24-percent lithium chloride solutions that are inhibited with 0.5-percent sodium dichromate and 0.2-percent oxalic acid.
2. A suitable pressure relief valve is recommended to insure safe operation below 500 psi.

## REFERENCES

1. NAVCERELAB Technical Memorandum M-108, "Evaluation of Halogenated Hydrocarbon and Alkali-Earth-Metal-Salt Fire Extinguishing Agents for Low Temperatures," 1 November 1955.
2. NAVCERELAB ltr NT4-59/NY 030 019-1.04, 200D5/CVB/bjs, ser 906 of 25 June 1956.
3. H. H. Uhlig, Handbook of Corrosion (John Wiley and Sons).

Table I. Corrosion Losses at 140°F (mdd)\*

Panel No.	Inhibitor	Test Panel **								
		B	B-1	B-2	C	C-1	C-2	S	S-1	S-2
1	0.2% Oxalic acid (all panels) (+ Mossy tin with B-1, C-1 and S-1 only)	6.06	4.83	15.98	8.55	14.33	6.86	1.26	4.02	1.32
2	0.2% p-Aminobenzoic acid (all panels) (+ Mossy tin with B-1, C-1 and S-1 only)	4.37	1.14	4.30	5.68	6.02	9.08	0.66	2.12	2.04
3	0.2% Diisopropyl amine	2.33	3.16	2.66	2.63	7.50	4.45	1.04	2.75	1.04
4	0.4% Diethyl amine	2.26	8.27	2.26	4.99	4.10	6.07	0.25	1.39	1.77
5 ***	0.1% coconut fatty acid nitriles, 0.5% Polyoxyethylene-alkyl ether, 0.1% Chlorinated biphenyl	4.16	27.53	2.68	5.05	14.26	7.80	2.59	2.02	3.35
6 ***	0.2% coconut fatty acid nitriles	4.02	5.97	4.52	6.11	11.56	5.08	0.58	1.41	0.83
7	0.3% Diethanolamine, 0.5% Sodium dichromate	0.28	1.20	0.28	1.64	1.45	1.60	0.06	0.19	0.12
8	0.3% Diethylethanolamine, 0.5% Sodium dichromate	0.14	1.13	0.21	1.45	1.58	1.67	0.44	0.13	0.12
9	0.3% Ethanolamine, 0.5% Sodium dichromate	1.20	2.00	1.41	1.64	1.96	2.40	0.25	0.88	0.19
10	0.5% Sodium dichromate	0.36	1.07	0.28	1.20	2.15	3.13	0.19	0.00	0.63
11	0.5% Sodium chromate	0.85	2.13	1.13	1.64	1.73	1.53	0.68	0.57	0.32
12	0.5% Sodium dichromate + sodium hydroxide to basic	0.68	1.94	0.89	1.53	1.59	1.55	0.24	0.55	0.49
13	0.5% Triethanolamine, 0.5% Sodium dichromate	0.34	0.71	0.23	0.92	1.28	1.48	2.02	0.05	0.12
14	0.2% Oxalic acid, 0.5% Sodium dichromate	0.21	0.39	0.27	0.80	2.02	0.71	0.61	0.24	0.00
15	0.2% Citric acid, 0.5% Sodium dichromate	0.27	0.39	0.26	1.90	0.67	0.77	0.18	0.55	0.30
16	Control (average)	2.39	10.26	3.64	6.20	8.15	4.36	1.18	4.09	1.71

\* mdd = milligrams per square decimeter per day

## \*\* Test Panels

B = Brass

B-1 = Brass + tin-lead solder along one edge

B-2 = Brass + silver solder along one edge

C = Copper

C-1 = Copper + tin-lead solder along one edge

C-2 = Copper + silver solder along one edge

S = Stainless steel

S-1 = Stainless steel + tin-lead solder along one edge

S-2 = Stainless steel plus silver solder along one edge

\*\*\* principally dodecyl nitrile.

Table II. Corrosion Losses at 100°F (mdd)\*

Panel No.	Inhibitor	Test Panel**								
		B	B-1	B-2	C	C-1	C-2	S	S-1	S-2
1	0.2% Oxalic acid (all panels) (+Mossy tin with B-1, C-1 and S-1 only)	1.34	2.67	1.48	1.86	3.67	11.04	0.42	1.80	0.84
2.	0.2% p-Aminobenzoic acid (all panels) (+ Mossy tin with B-1, C-1 and S-1 only)	1.14	1.97	1.28	1.80	1.69	1.59	0.12	0.66	0.60
3	0.2% Diisopropyl amine	1.85	2.90	6.35	2.64	2.07	4.33	0.18	0.79	0.43
4	0.4% Diethyl amine	2.05	1.70	6.36	1.20	1.52	2.33	0.25	1.07	0.00
5 ***	0.1% coconut fatty acid nitriles, 0.05% Polyoxyethylene-alkyl ether, 0.1% Chlorinated biphenyl	1.41	2.53	1.20	1.58	1.77	1.40	0.35	0.44	0.51
6 ***	0.2% coconut fatty acid nitriles	5.32	9.15	1.58	1.35	2.18	1.15	0.19	0.83	0.90
7	0.3% Diethanolamine, 0.5% Sodium dichromate	0.35	1.07	0.28	4.86	2.35	6.47	0.11	0.38	0.06
8	0.3% Diethylethanolamine, 0.5% Sodium dichromate	0.35	1.40	0.28	1.45	2.91	1.13	0.11	0.25	0.06
9	0.3% Ethanolamine, 0.5% Sodium dichromate	7.21	4.47	7.71	2.02	7.34	8.60	0.06	0.63	0.25
10	0.5% Sodium dichromate	0.42	2.73	0.42	0.82	6.11	2.20	0.11	0.38	0.19
11	0.5% Sodium chromate	7.14	5.80	7.71	2.34	2.34	3.13	0.11	0.38	0.13
12	0.5% Sodium dichromate + sodium hydroxide to basic	4.65	2.26	2.74	1.65	6.30	3.38	0.11	0.55	0.00
13	0.5% Triethanolamine, 0.5% Sodium dichromate	1.78	0.90	0.27	1.71	6.56	1.74	0.18	0.00	0.18
14	0.2% Oxalic acid, 0.5% Sodium dichromate	0.41	0.71	0.41	1.28	1.47	0.84	0.00	0.00	0.06
15	0.2% Citric acid, 0.5% Sodium dichromate	0.27	0.19	0.41	1.10	2.26	1.03	0.00	0.37	0.00
16	Control (average)	1.05	2.80	1.21	1.57	3.29	2.34	0.18	1.53	0.57

\* mdd = milligrams per square decimeter per day

\*\* Test Panels

B = Brass

B-1 = Brass plus tin-lead solder along one edge

B-2 = Brass plus silver solder along one edge

C = Copper

C-1 = Copper plus tin-lead solder along one edge

C-2 = Copper plus silver solder along one edge

S = Stainless Steel

S-1 = Stainless steel plus tin-lead solder along one edge

S-2 = Stainless steel plus silver solder along one edge

\*\*\* Principally dodecyl nitrile



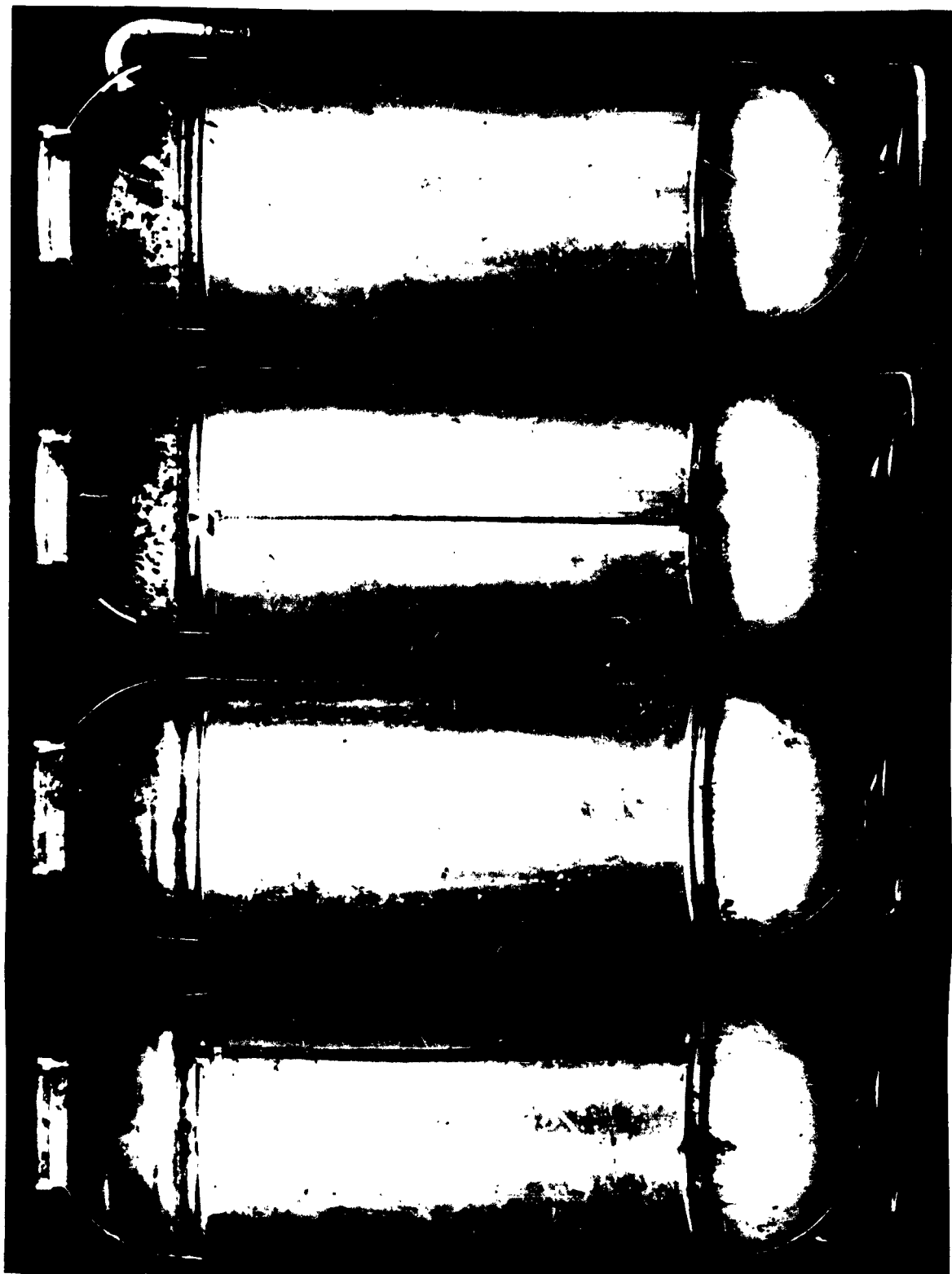


Figure 1. Stainless steel Extinguishers Nos. 1 and 2, Inside View.  
Austenitic Inconel holes formed by corrosion



Figure 2. Stainless Steel Extinguisher No. 3.

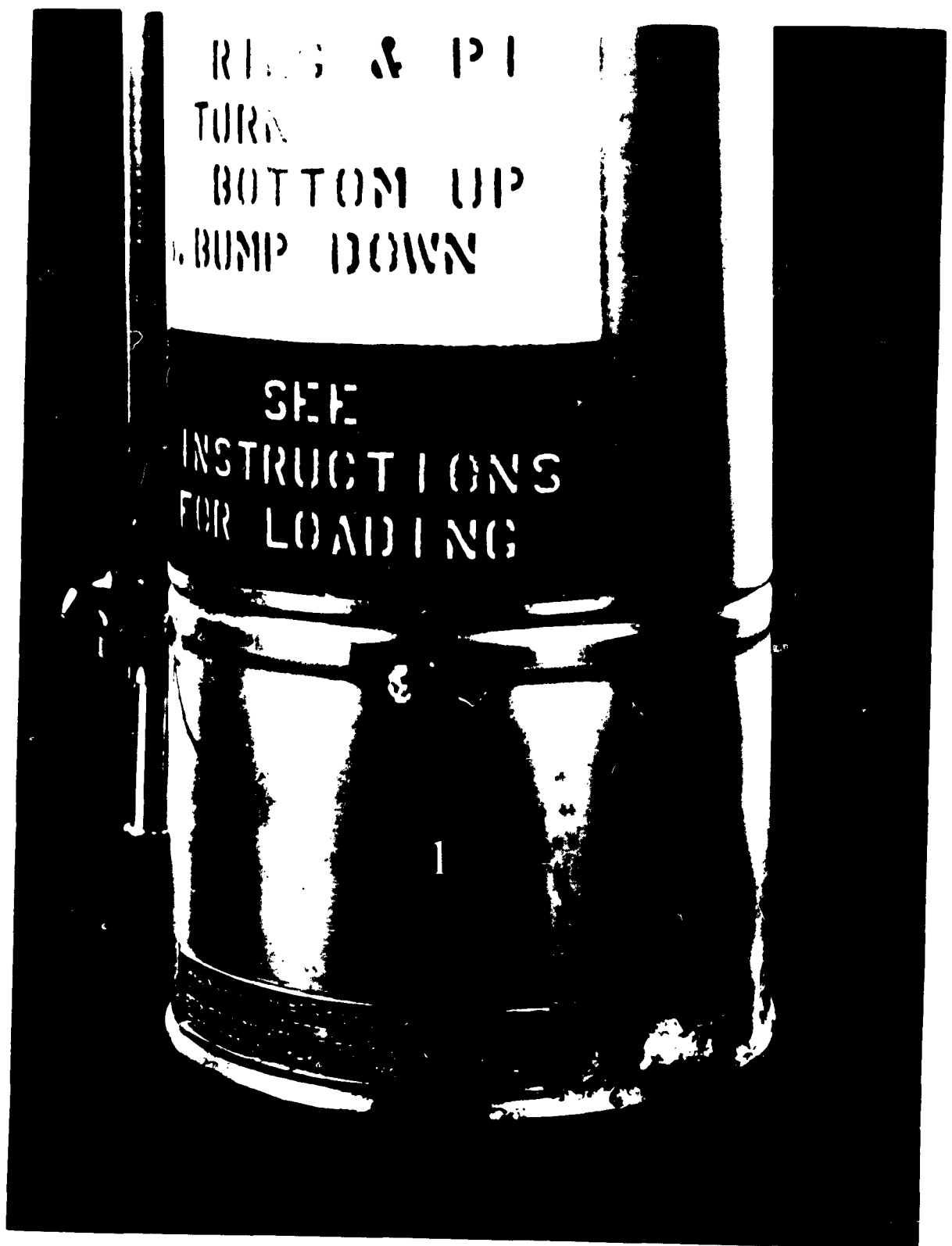


Figure 3. Stainless Steel Extinguisher No. 1, Outside View.  
Arrows locate holes formed by corrosion.

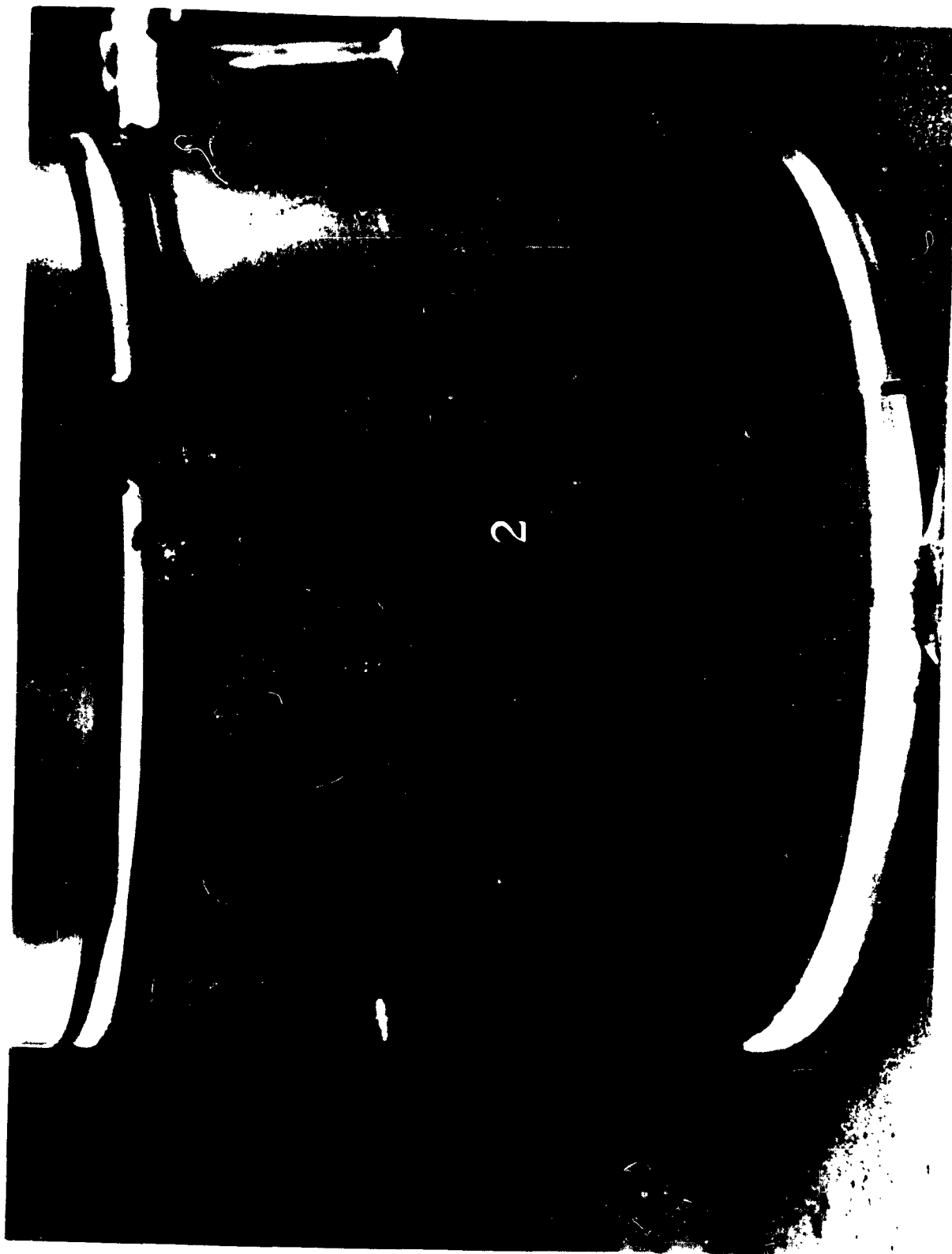


Figure 4. Stainless Steel Extinguisher No. 2, Outside View.  
Arrow locates holes formed by corrosion.



Figure 5. Stainless Steel Extinguisher No. 1, Bottom Inside.  
Arrows locate holes formed by corrosion.



Figure 6. Stainless Steel Extinguisher No. 2, Bottom Inside.  
Arrow locates hole formed by corrosion.

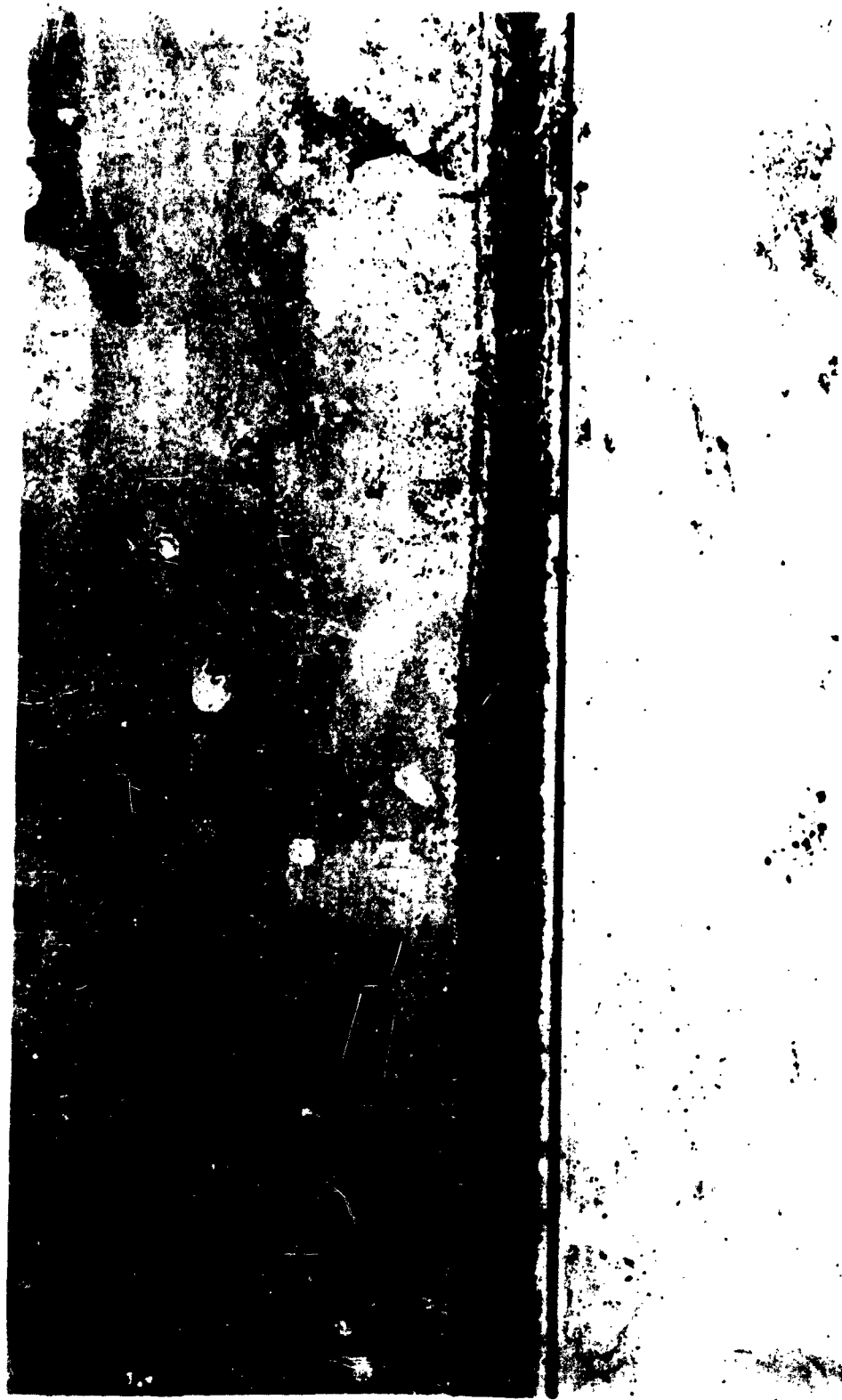


Figure 7. Stainless Steel Extinguisher No. 1, Inside  
Intergranular corrosion along weld.

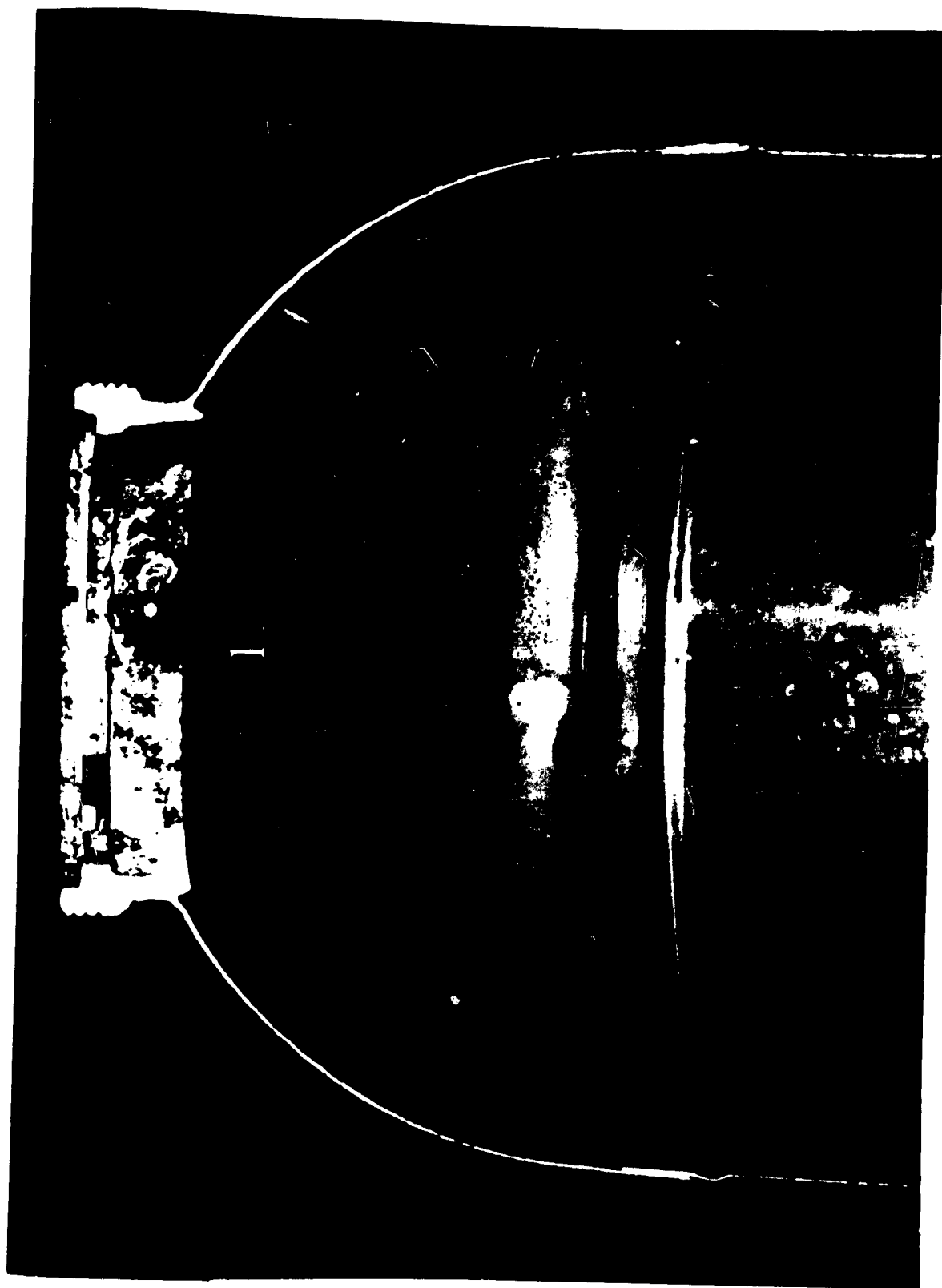


Figure 8. Stainless Steel Extinguisher No. 1, Top Inside. Greasy carbon film (right half) and clean metal after removal of film (left half).





Figure 9. Copper Extinguisher No. 4, Inside View.



Figure 10. Copper Extinguisher No. 5, Inside View.



Figure 11. Copper Extinguisher No. 6, Inside View.

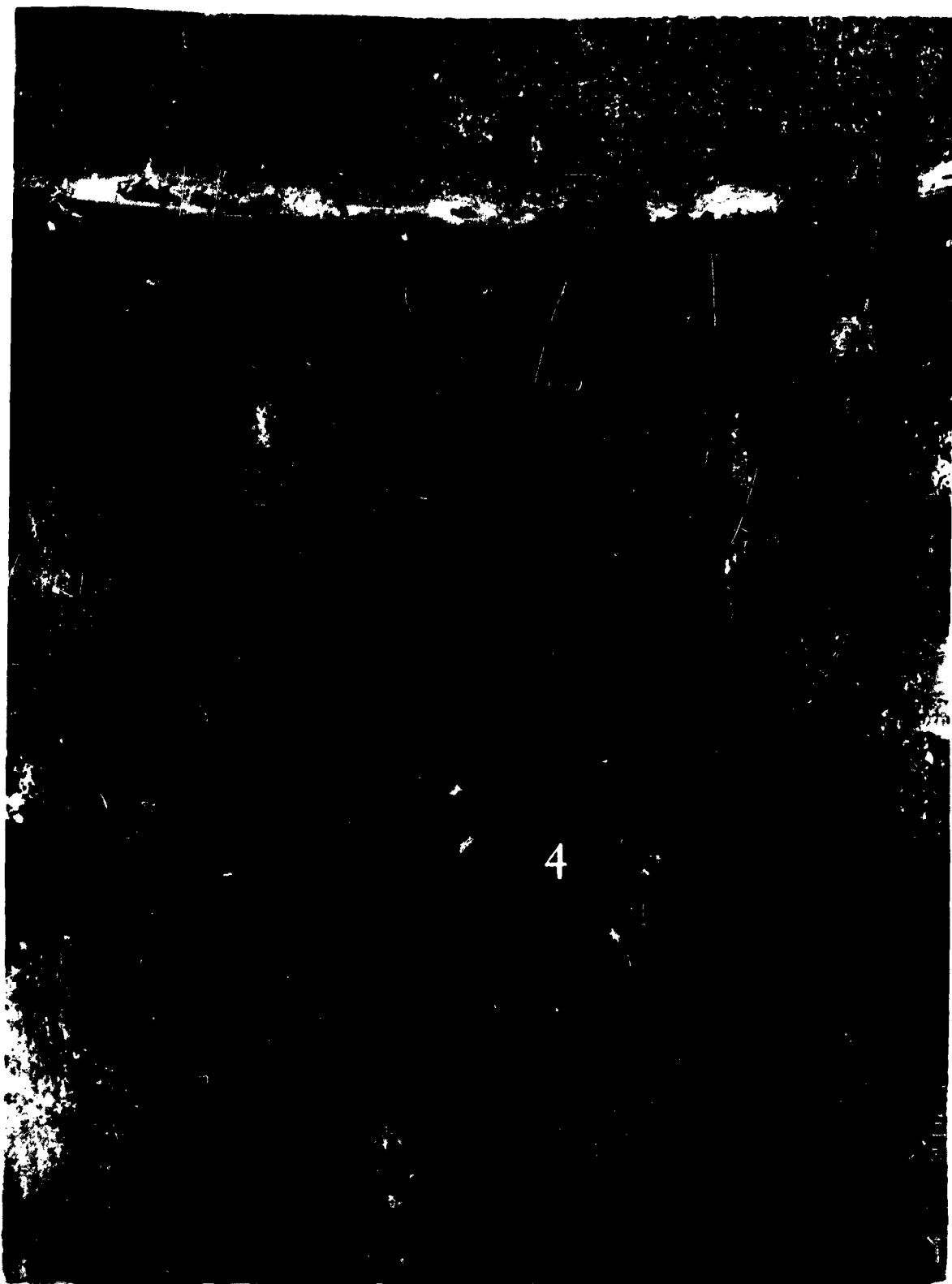


Figure 12. Copper Extinguisher No. 4, Inside View. Corroded solder showing porous lead residue and lead chromate crystals.